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IS 12308-9 (1993): Methods for Chemical Analysis of Cast Iron and Pig Iron, Part 9: Determination of Molybdenum By Thiocyanate (Spectrometric) Method (for Molybdenum 0.01 to 1. [MTD 6: Pig iron and Cast Iron]

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IS 12308 (Part 9) : 1993
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Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF CAST IRON AND PIG IRON

PART 9 DETERMINATION OF MOLYBDENUM BY THIOCYANATE (SPECTROMETRIC) METHOD (FOR MOLYBDENUM 0·01 TO 1·0 PERCENT)

UDC 669·162·275·12 + 669·13 : 543·42 [546·77]

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

December 1993 **Price Group 1**

Methods of Chemical Analysis of Ferrous Metals Sectional Committee, MTD 2

FOREWORD

This Indian Standard (Part 9) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

Chemical analysis of cast iron and pig iron was covered in IS 228 : 1959 'Methods of chemical analysis of pig iron, cast iron and plain carbon and low alloy steels (*revised*)'. During its second revision it was decided that a comprehensive series should be prepared for chemical analysis of all types of steels and the other covering the chemical analysis of cast iron and pig iron. Accordingly IS 228 on revision was published in several parts covering chemical analysis of various steels only and a separate series of standards under IS 12308 is being published for chemical analysis of cast iron and pig iron. This standard (Part 9) is one in the latter series. The other parts in the series are:

IS 12308 Methods of chemical analysis of cast iron and pig iron

- Part 1 Determination of total carbon by thermal conductivity method
- Part 2 Determination of sulphur by iodimetric titration method
- Part 3 Determination of manganese by periodate spectrophotometric method
- Part 4 Determination of total carbon graphitic, carbon and combined carbon by gravimetric method
- Part 5 Determination of phosphorus (0·01 to 0·50 percent) by alkalimetric method
- Part 6 Determination of silicon (for silicon 0·1 to 6·0 percent)
- Part 7 Determination of nickel by dimethylglyoxime (gravimetric) method (for nickel 0·5 to 36 percent)
- Part 8 Determination of chromium by persulphate oxidation method (for chromium 0·1 to 28 percent)
- Part 10 Determination of manganese (up to 7·0 percent) by arsenite (volumetric) method
- Part 11 Determination of total carbon by the direct combustion volumetric method (for carbon 1·50 to 4·50 percent)
- Part 12 Determination of copper by atomic absorption spectrometric method (for copper 0·01 to 0·5 percent)
- Part 13 Determination of magnesium by atomic absorption spectrometric method (for magnesium up to 0·1 percent)

In this revision the thiocyanate spectrometric method for determination of molybdenum in pig iron and cast iron has been updated on the basis of experience gained during the past and reproducibility of the method has been incorporated at different percent levels.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*).'

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF CAST IRON AND PIG IRON

PART 9 DETERMINATION OF MOLYBDENUM BY THIOCYANATE (SPECTROMETRIC) METHOD (FOR MOLYBDENUM 0·01 TO 1·0 PERCENT)

1 SCOPE

This standard (Part 9) describes the method for determination of molybdenum in cast iron and pig iron in the range from 0·01 to 1·0 percent.

2 REFERENCE

IS No.	*Title*
264 : 1976	Nitric acid (*second revision*)
266 : 1977	Sulphuric acid (*second revision*)
1070 : 1992	Reagent grade water (*third revision*)

3 SAMPLING

Samples shall be drawn and prepared as prescribed in the relevant Indian Standard.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water (*see* IS 1070 : 1992) shall be employed for the test.

5 DETERMINATION OF MOLYBDENUM

5.1 Outline of the Method

An aliquot of the sample solution is treated with potassium thiocyanate to develop the molybdenum and iron thiocyanate complex and reduced with stannous chloride. Molybdenum complex is extracted with *n*-Butyl acetate and measured at 470 nm.

5.2 Reagents

5.2.1 *Sulphuric Acid—Phosphoric Acid Mixture*

Add slowly, while stirring 160 ml of sulphuric acid (rd = 1·84) to 760 ml of water. Cool and add 80 ml of phosphoric acid.

5.2.2 *Nitric Acid*, rd = 1·42 (Conforming to IS 264 : 1976).

5.2.3 *Dilute Sulphuric Acid*, 1 : 5 (*v/v*)

5.2.4 *Hydrofluoric Acid*, 40 percent

5.2.5 *Potassium Bisulphate*, solid

5.2.6 *Potassium Thiocyanate*, 10 percent (*m/v*)

5.2.7 *Stannous Chloride Solution*, 300 g/l

Dissolve 60 g of stannous chloride in 20 ml hydrochloric acid and heat at 60-70°C to dissolve completely. Cool and dilute to 200 ml with water. Add a few pieces of metallic tin and store in a stoppered bottle.

5.2.8 *N-Butyl Acetate*

5.2.9 *Iron*, molybdenum free

5.2.10 *Ammonia Solution*, rd = 0·90

5.2.11 *Dilute Hydrochloric Acid*, 1 : 1 (*v/v*)

5.2.12 *Standard Molybdenum Solution* (*1 ml = 0·1 mg* Mo)

Transfer 0·100 0 g of molybdenum metal (99·9 percent pure) to a 250 ml conical flask and dissolve it with 10 ml of hydrochloric acid and 10 ml water. Add 1 ml of nitric acid and heat to dissolution. Cool, dilute to one litre with water in a volumetric flask.

5.2.12.1 *Standard molybdenum solution* (*1 ml = 0·01 mg* Mo)

Take 100 ml of this solution and dilute to one litre in a volumetric flask with water.

Alternatively

Dissolve 0·150 0 g of molybdenum trioxide (MoO_3) in minimum amount of ammonia solution. Acidify with dilute hydrochloric acid (1 : 1) and dilute to one litre in a volumetric flask with water. Take 100 ml of this solution and dilute to one litre with water in a volumetric flask.

5.3 Procedure

5.3.1 Transfer 0·500 g of the sample to a 150 ml beaker. Add 50 ml of the acid mixture and heat gently to dissolve the sample. Add nitric acid dropwise till no further brown fumes are formed. Evaporate the solution to light fuming. Cool, dilute to 50 ml with water and heat to dissolve the salts. Filter the solution through a medium textured filter paper washing with hot water 8 to 10 times. Preserve the filtrate. Transfer the residue with filter paper in a platinum crucible. Ignite the residue at about 600°C. Cool and moisten with few drops of dilute sulphuric acid (1 : 5) and 4 to 5 ml hydrofluoric acid. Evaporate till fumes of sulphur trioxide disappear and fuse the residue with potassium bisulphate and take up the melt in dilute sulphuric acid. Mix this solution with the preserved filtrate and dilute to 250 ml in a volumetric flask and mix well.

5.3.2 Pipette out a suitable aliquot containing 0·05-0·10 mg molybdenum to a 250 ml separating funnel. Add in sequences, 15 ml of potassium thiocyanate and 15 ml of stannous chloride solution. Mix thoroughly after each addition. Add 25 ml of *n*-Butyl acetate, stopper and shake vigorously for one minute. Allow the phases to separate. Remove the stopper and drain out the aqueous phase. Add to the organic phase 50 ml of dilute sulphuric acid, 5 ml of potassium thiocyanate and 5 ml of stannous chloride solution. Stopper and shake vigorously for one minute. Allow the phases to separate. Drain off the aqueous layer and discard.

5.3.3 Collect the organic layer after passing through a dry filter paper. Transfer a suitable portion of this to a 1-cm cell and measure the absorbance at 470 nm against the reagent blank.

5.3.4 Convert the photometric readings of test solution to mg of molybdenum obtained from calibration curve and calculate the percentage of molybdenum as given in **5.3.7**.

5.3.5 *Blank*

Carry out a blank along with the test sample using all the reagents and 0·5 g iron instead of the sample and proceed according to the method specified in **5.3.1** to **5.3.4**. Take the aliquot of butyl acetate for reagent blank.

5.3.6 *Preparation of the Calibration Curve*

Take 0·500 g of iron in each of eight 250-ml conical flasks. Transfer 0, 1, 2, 3, 4, 6, 8 and 10 ml of standard molybdenum solution (1 ml = 0·01 mg Mo) (*see* **5.2.12**) to the flasks. Add 50 ml of the acid mixture and proceed according to **5.3.1** to **5.3.3**. The flask containing the zero ml of molybdenum solution is regarded as blank and the net values of photometric reading is plotted against mg of molybdenum.

5.3.7 *Calculation*

$$\text{Molybdenum, percent by mass} = \frac{A}{B} \times \frac{1}{10}$$

where

A = mass, in mg, of molybdenum in the aliquot of the solution; and

B = mass in, g of the sample represented by the aliquot taken.

5.4 Reproducibility

±0·003 at 0·05 percent molybdenum,

±0.06 at 0·5 percent molybdenum, and

±0·07 at 1 percent molybdenum.

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